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# Comparison of Methods for Determining the Ferrite Content in Duplex Cast Steels

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## Abstract

The paper is concerned with comparing the methods for determining the ferrite content in castings from duplex stainless steels. It uses Schaeffler diagram, empirical formula based calculation, image analysis of metallographic sample, X-ray diffraction and measurement with a feritscope. The influence of wall thickness of the casting on the ferrite content was tested too. The results of the experiments show that the casting thickness of 25 or 60 mm does not have a significant effect on the measured amount of ferrite. The image analysis of metallographic sample and the measurement with the feritscope appear to be the most suitable methods. On the contrary, predictive methods, such as Schaeffler diagram or empirical formula based calculation are only indicative and cannot replace the real measurements. X-ray diffraction seems to be the least suitable measuring method. Values of ferrite content measured in such a way often deviated from the values measured by image analysis and with feritscope.

**Keywords:** Non-destructive testing, Duplex cast steel, Metallographic evaluation, Feritscope, XRD

## 1. Introduction

Duplex stainless steels were first described by Bain and Griffiths in 1927 but their practical use started in 1930's. They are high alloy chromium-nickel-molybdenum cast steels with a structure composed of two phases of ferrite and austenite. The result of this combination is excellent mechanical properties with excellent corrosion resistance in various corrosive environments. Ferrite is a carrier of the steel strength and it also guarantees its good weldability. Austenite then ensures good ductility and toughness. Important is also the fact that, compared to the austenitic steels, the duplex cast steels contain significantly less expensive nickel, which is positively reflected in their lower price. Thanks to these excellent properties, the duplex cast steels are used more and more often in practice and often replace other types of stainless steels. They are used for components working in sea water, at the extraction of oil and natural gas, in manufacture of paper, in power engineering, manufacture, transport and storage of chemicals, and recently more and more frequently in the construction of buildings and bridges [1, 2].

The content of the main structural components of steel, i.e., the ferrite and austenite, is important for achieving the desired mechanical and other properties. Steels containing 30 to 70 percent of ferrite are used in practice, but the most common are materials containing 50 % of ferrite or with a slight excess of austenite for achieving better strength-plastic properties [3].

Several different methods can be used to determine the ferrite content in steel structure. The easiest and only very approximate method is to use one of the constitution diagrams to estimate the resulting structure on the basis of the chemical composition of steel. Among the commonest ones is the Schaeffler diagram, which, in spite of the fact that it was introduced as early as 1949, is still widely used. Originally it was designed for the purposes of welding, but it can also be used for predicting the ferrite content

in steel in a cast condition. $Cr_{eq}$ and $Ni_{eq}$ are calculated by formulas (1) and (2) being inserted in the diagram [4].

$$Cr_{eq} = \%Cr + \%Mo + 1.5 \times \%Si + 0.5 \times \%Nb \quad (1)$$

$$Ni_{eq} = \%Ni + 0.5 \times \%Mn + 30 \times \%C + 30 \times \%N \quad (2)$$

Another method for the prediction of the resulting structure of duplex cast steel is a calculation based on an empirical equation, e.g., (3). The below equation also considers the influence of heat treatment, solution annealing, which is almost always used for castings of duplex stainless steels [3].

$$\%F = -20.93 + 4.01 \times Cr_{eq} - 5.6 \times Ni_{eq} + 0.016 \times T \quad (3)$$

where [3]:

$$Cr_{eq} = \%Cr + 1.73 \times \%Si + 0.88 \times \%Mo \quad (4)$$

$$Ni_{eq} = \%Ni + 24.55 \times \%C + 21.75 \times \%N + 0.4 \times \%Cu \quad (5)$$

T [°C] is temperature of temper annealing in the range of 1050-1150°C.

It should be pointed out here that $Ni_{eq}$ and $Cr_{eq}$ according to equations (4) and (5) differ from $Cr_{eq}$ and $Ni_{eq}$ according to equations (1) and (2) and cannot be confused.

These were the methods used for predicting the steel structure. More important, however, are methods that enable the direct measurement of ferrite content in the real material. These include:

- **image analysis of metallographic sample** – this is a destructive method, where a sample must be cut from the tested material, a metallographic ground must be prepared, photos of the structure using an optical microscope must be taken and then they must be evaluated using the methods of image analysis.
- **X-ray diffraction** – a beam of monochromatic X-ray radiation is utilized here. When the X-ray irradiation passes through the crystalline material, the diffraction of beams occurs on the crystal planes. The present structural phases and their share can then be determined from the directions and intensities of beams of the diffracted irradiation. The measurement is performed to the depth of a few micrometers.
- **Feritscope** – a device based on the magneto-inductive method. The probe consists of two coils with one inducing an electromagnetic field that enters the tested material to a depth of approximately 1-2 mm where it interacts with ferromagnetic phases. These changes in the magnetic field produce electrical voltage in the second coil that is proportional to the content of the ferromagnetic phase, in this case, of ferrite [5].

All of these methods make it possible to detect the ferrite content in duplex stainless steels, but each works on a completely different principle. Thus, the aim of this paper is to compare the results of the measurements obtained by individual methods, and compare their advantages and disadvantages. The influence of wall thickness of the casting will be tested, too.

Duplex stainless steels are produced as castings and wrought semi-finished products. This paper is devoted to the problems of cast materials.

## 2. Conditions of the experiment and methods used

For the purposes of this experiment, the castings were chosen from six different melts of the duplex stainless steel cast in the foundry of Brno University of Technology. The samples are marked with a melt number with their chemical composition measured by 0the optical emission spectrometer ARL 4460 being shown in Table 1. The nitrogen content was determined using the analyzer LECO TC 600.

Table 1.
Chemical composition of experimental melts in wt. %

| Sample number | C | Mn | Si | Cr | Ni | Mo | N |
|---|---|---|---|---|---|---|---|
| 243 | 0.02 | 0.95 | 0.28 | 21.00 | 3.70 | 1.70 | 0.1110 |
| 244 | 0.02 | 0.94 | 0.27 | 23.55 | 6.05 | 3.10 | 0.1636 |
| 246 | 0.02 | 0.88 | 0.30 | 23.70 | 5.50 | 3.10 | 0.1719 |
| 247 | 0.03 | 1.00 | 0.26 | 21.30 | 3.60 | 1.65 | 0.1085 |
| 248 | 0.02 | 0.98 | 0.30 | 20.50 | 8.30 | 4.60 | 0.1268 |
| 249 | 0.02 | 1.00 | 0.26 | 26.80 | 3.80 | 1.75 | 0.2657 |

To verify the influence of the casting wall thickness on the ferrite amount in the structure, the test castings of Y-blocks with thicknesses of 25 mm and 60 mm were produced from each of these melts. The material was melted in a vacuum electric induction furnace with a neutral lining and maximum melt weight of 120 kg. The melt was vacuumed for 20 minutes at a temperature of 1600°C and pressure of 2500 Pa in order to reduce the carbon content, to reduce the amount of gases and oxidic inclusions. To improve the course of the carbon reaction, the melt was refined with argon through a porous plug in the furnace bottom. The melt was cast in moulds of quartz base sand bound with alkaline phenol resin Alphaset. The results were two castings of different wall thicknesses from each melt, totally 12 test samples. After casting, these castings were heat treated by solution annealing at a temperature of 1130°C lasting 5 hours and then cooled in water. Samples are marked with the melt number plus Y25 or Y60, according to the casting wall thickness.

According to Figure 1, the samples for the preparation of test bars for the tensile test were cut from these test Y-blocks. As the evaluation of the mechanical properties was not the aim of this experiment, the results of the tensile tests are not given here. After the rupture of the test bars, metallographic samples were made from the die heads using common grinding and polishing wet methods. They were also used for determining the ferrite content using all the above methods.

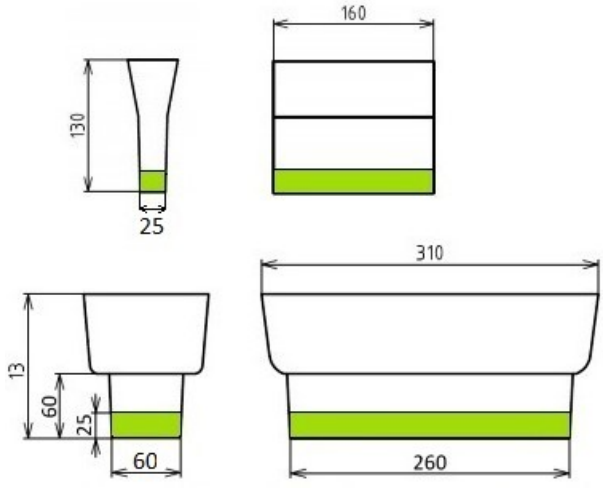


Fig. 1. Test castings

Image analysis was performed on samples etched with the Beraha II etching agent when the austenite remains white, while the ferrite is coloured black. The LECO IA32 program was used for the actual analysis. On each sample, 20 observations and evaluations of structure were carried out. The following values are the averages of these twenty measurements.

Measurements using X-ray diffraction were carried out on the diffractometer XPert Panalytical in the Bragg-Brentan arrangement, using Cu-Kα radiation. Before measuring, the samples were etched for 5 minutes in the etching agent 80% $H_2O_2$ + 20% HF in order to remove the surface layer of crystals deformed by polishing.

For measurements using the magneto-inductive method a Feritscope Fisher FMP30 was used. Ten measurements were carried out on each of the samples; the results are their arithmetic means.

# 3. Measurement results and discussion

Below, the results will be listed of predicting and measuring the ferrite content in experimental samples by using individual methods described above.

## 3.1 The Schaeffler diagram

The Schaeffler diagram can be used for an approximate prediction of the resulting casting structure. $C_{req}$ and $Ni_{eq}$ were calculated from the chemical composition of cast melts by Table 1 using formulas (1) and (2) with their values subsequently plotted

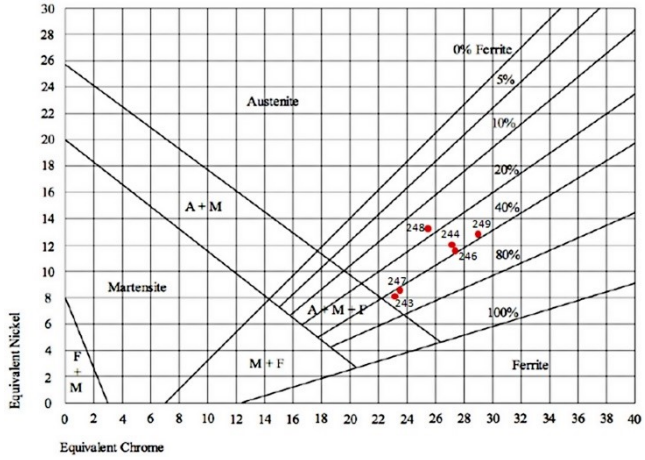


Fig. 2. Position of experimental melts in the diagram (Schaeffler diagram taken from [6])

in the diagram in Figure 2. As can be seen here, the lowest ferrite content can be estimated in the melt 248, namely, about 17 %. In contrast, the highest ferrite content around 50 % is predicted by the diagram for the melt 243 and, similarly, for melt 247, too. For the remaining melts 244, 246 and 249, about 40 % of ferrite can be estimated in their structures. Table 2 provides a clearer view of these data. The numbers shown are only subjective readings of the diagram shown in Figure 2.

Table 2.
Ferrite content according to the Schaeffler diagram

| sample | 243 | 244 | 246 | 247 | 248 | 249 |
|---|---|---|---|---|---|---|
| %F | 50 | 35 | 40 | 50 | 17 | 37 |

## 3.2 Formula-based calculation

Another option used for estimating the ferrite content in individual melts is the calculation by formula (3). The results are listed in Table 3.

Table 3.
Resulting ferrite contents by formula (3)

| sample | 243 | 244 | 246 | 247 | 248 | 249 |
|---|---|---|---|---|---|---|
| %F | 51.8 | 46.8 | 49.7 | 52.2 | 31.5 | 55.6 |

It can be seen that, although the calculation shows the trend of the lowest ferrite content in structure of the sample 248, i.e. similarly to the Schaeffler diagram, the numerical values of the ferrite contents are often significantly different (see e.g. samples 248 or 249).

## 3.3 Imageanalysis

A method, which is very frequently used in practice, is the image analysis of a metallographic sample. Here it is the real

measurements on real samples that matter rather than an estimation or calculation. The time demands for preparing the metallographic sample and subsequent image analysis are the disadvantages. Structure of the sample 248-Y60, i.e., the sample with the lowest measured ferrite content, is shown in Figure 3 as an example. By contrast, Figure 4 shows the structure of the sample 249-Y25 with the highest registered ferrite content.

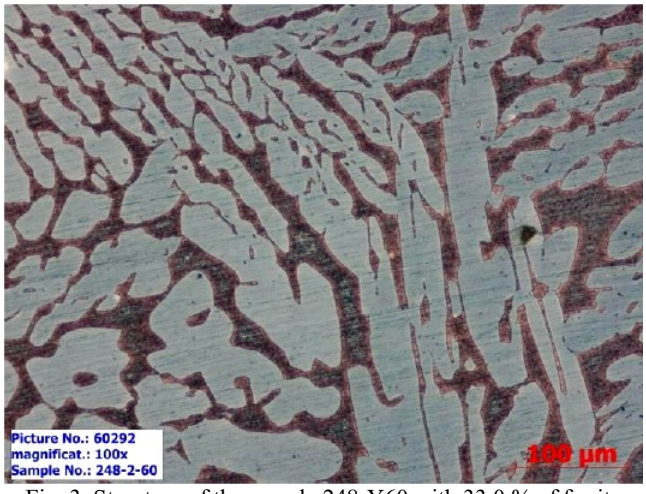


Fig. 3. Structure of the sample 248-Y60 with 33.0 % of ferrite

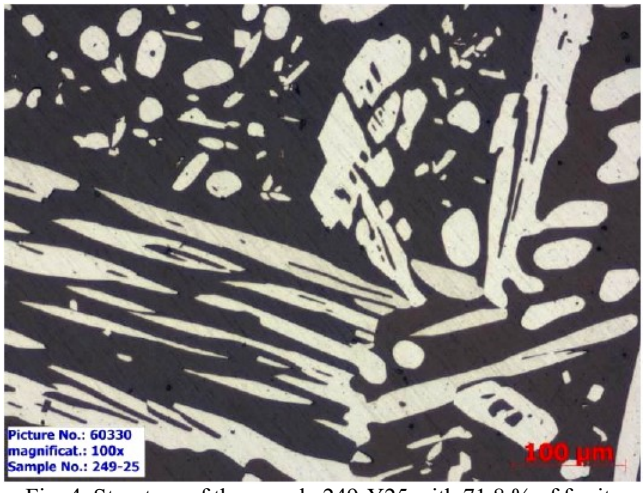


Fig. 4. Structure of the sample 249-Y25 with 71.8 % of ferrite

All ferrite contents determined by image analysis of samples from the castings with wall thicknesses of 25 and 60 mm are shown in Table 4 along with standard deviations (σ) determined by the image analysis program.

Table 4.
Ferrite content determined using the image analysis

| sample | %F | σ | sample | %F | σ |
|---|---|---|---|---|---|
| **243-Y25** | 52.7 | 3.14 | **243-Y60** | 54.0 | 4.53 |
| **244-Y25** | 55.3 | 1.22 | **244-Y60** | 60.2 | 2.61 |
| **246-Y25** | 61.4 | 5.01 | **246-Y60** | 61.0 | 0.86 |
| **247-Y25** | 60.4 | 1.69 | **247-Y60** | 57.2 | 6.77 |
| **248-Y25** | 37.4 | 5.58 | **248-Y60** | 33.0 | 1.46 |
| **249-Y25** | 71.8 | 1.47 | **249-Y60** | 68.8 | 3.50 |

It can be seen from the results that, when using the image analysis, no substantial difference in the ferrite content depending on the wall thickness of the casting has been determined. With regard to the standard deviations values, the differences in the ferrite content measured in both wall thicknesses are negligible.

## 3.4 X-ray diffraction

Another method used in practice for measuring the ferrite content in duplex stainless steels is X-ray diffraction. The time demand is a disadvantage of this method; the measurement takes several hours. Another disadvantage is the need to use a very expensive device, the diffractometer, whose operation is relatively complex requiring a high expertise of the operator.

Table 5.
Ferrite content determined using X-ray diffraction

| sample | %F | sample | %F |
|---|---|---|---|
| **243-Y25** | 73.7 | **243-Y60** | 72.5 |
| **244-Y25** | 54.4 | **244-Y60** | 29.5 |
| **246-Y25** | 54.3 | **246-Y60** | 50.4 |
| **247-Y25** | 63.5 | **247-Y60** | 71.5 |
| **248-Y25** | 34.3 | **248-Y60** | 38.9 |
| **249-Y25** | 61.4 | **249-Y60** | 60.7 |

It can be seen again from the measurement results that the differences between the ferrite content in castings with wall thicknesses of 25 mm and 60 mm are not striking. An exception is the sample from the melt 244 where, for the sample 244-Y25, 54.4 % of ferrite was measured while, in the sample 244-Y60, it was 29.5 % only. But such low ferrite content does not correspond either to the results of the image analysis or to the below measurement using the feritscope. Most likely, this is due to a measurement error.

## 3.5 Measurements with the feritscope

Feritscope is a device that is more frequently used in practice for the structural analyses of welds because the necessary equipment is easy to carry and it almost enables spot measuring even in less accessible places. However, equally well it can also be used for determining ferrite content in duplex stainless steels.

The results of measurements on the experimental samples are shown in Table 6.

Table 6.
Ferrite content measured by feritscope

| sample | %F | σ | sample | %F | σ |
|---|---|---|---|---|---|
| **243-Y25** | 67.4 | 1.4 | **243-Y60** | 66.7 | 2.6 |
| **244-Y25** | 54.5 | 2.0 | **244-Y60** | 55.9 | 2.4 |
| **246-Y25** | 60.5 | 2.2 | **246-Y60** | 60.3 | 1.5 |
| **247-Y25** | 65.9 | 1.6 | **247-Y60** | 64.8 | 2.3 |
| **248-Y25** | 33.6 | 1.7 | **248-Y60** | 36.0 | 1.5 |
| **249-Y25** | 65.8 | 1.7 | **249-Y60** | 62.4 | 3.0 |

Not even measurements with the feritscope show more substantial differences in the ferrite content between casting samples with wall thicknesses of 25 or 60 mm.

## 3.6 Summary of results

In order to better compare the results of individual methods, the values measured are summarized in Table 7. For easier visual comparison, these values are then also displayed graphically in Figure 5. It can be seen that the estimates of the ferrite content from the Schaeffler diagram or calculated by Formula (3) are only approximate and can be burdened by a significant error. For more precise determination of the ferrite content in the steel, they are, therefore, absolutely inappropriate.

Table 7.
Summarized measured data

| sample | Y25 | | | Y60 | | | calculation | Schaeffler |
|---|---|---|---|---|---|---|---|---|
| | image analysis | XRD | feritscope | image analysis | XRD | feritscope | | |
| | %F | %F | %F | %F | %F | %F | %F | %F |
| **243** | 52.7 | 73.7 | 67.4 | 54.0 | 72.5 | 66.7 | 51.8 | 50 |
| **244** | 55.3 | 54.4 | 54.5 | 60.2 | 29.5 | 55.9 | 46.8 | 35 |
| **246** | 61.4 | 54.3 | 60.5 | 61.0 | 50.4 | 60.3 | 49.7 | 40 |
| **247** | 60.4 | 63.5 | 65.9 | 57.2 | 71.5 | 64.8 | 52.2 | 50 |
| **248** | 37.4 | 34.3 | 33.6 | 33.0 | 38.9 | 36.0 | 31.5 | 17 |
| **249** | 71.8 | 61.4 | 65.8 | 68.8 | 60.7 | 62.4 | 55.6 | 38 |

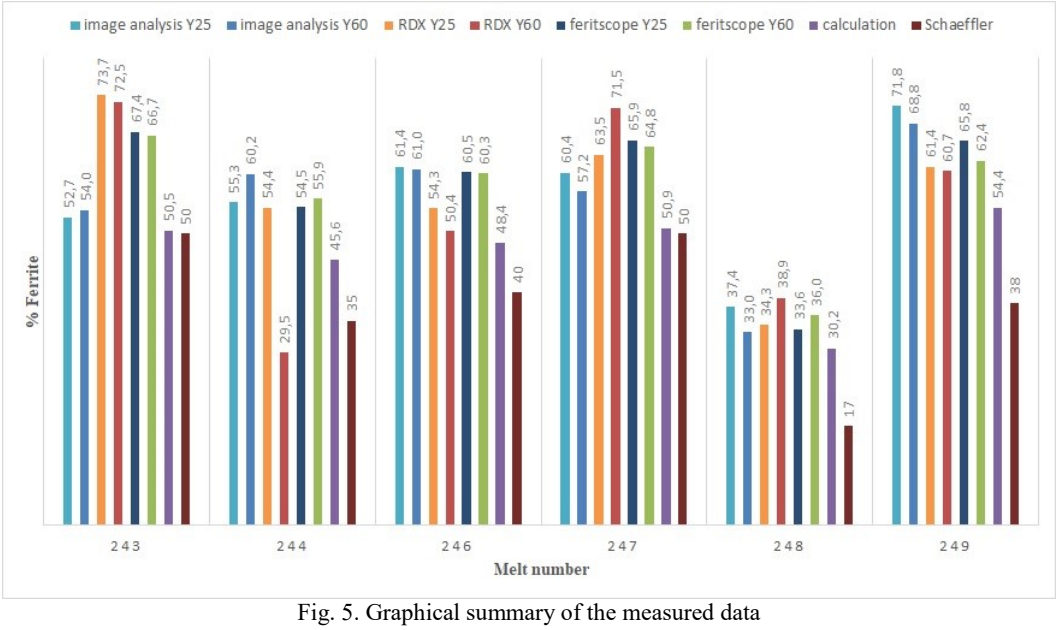


Fig. 5. Graphical summary of the measured data

Therefore, we will only deal with the methods of measurement on a real sample. It is, however, also apparent that the ferrite contents determined by different methods on the same sample are often significantly different. The most striking difference is the one for the sample 243-Y25 where, using the image analysis, the ferrite content of 52.7 % was detected while by the X-ray diffraction it was 73.7 %. This means a difference of 21 %! On the contrary, a minimum difference of 0.9 % was registered in the sample 244-

Y25 where the results of all three measuring methods were exceptionally balanced. These were the two extremes, but for the other samples, the differences are not negligible. It might be concluded that the differences between the minimum and maximum values of ferrite content measured by different methods on one sample range around 10 %.

# 4. Conclusions

The results of the experiments with the measurements of ferrite content in duplex stainless steels using a variety of methods can be summarized as follows:

- predictive methods, such as Schaeffler diagram or the calculations based on empirical formulas are merely approximate and cannot replace the real measurements.
- no significant difference in the ferrite content for castings with wall thicknesses of 25 and 60 mm was observed regardless of the method used. Only in a casting with a thicker wall there is a slight increase of the variance of individual measurements for the same sample and, thereby, the increase of the values of standard deviations. It may be due to the increasing grain size in castings with increasing wall thickness. All of the above measuring methods have a relatively small volume of the tested material (max 1-2 mm) and, therefore, with increasing grain size, the measurements will vary more from place to place.
- the image analysis of a metallographic sample is often used in practice and gives fairly good results. It is, however, necessary to make a sufficient number of measurements over a greater area of the sample. The place in which a measurement was made, i.e., the grain size and the direction of grain growth is of importance as well. This is an areal method and, therefore, it is cannot consider the material volume. The method is quite time demanding. It is an advantage that the metallographic sample can further be used for the evaluation of grain size, the presence of intermetallic phases, etc.
- in this case the X-ray diffraction seems to be the least suitable method. The values of ferrite content measured in this way often deviate from the values measured by image analysis and with a feritscope. In the case of sample 244-Y60, the measurement was even wrong. The problem is in a small depth of radiation penetration into the investigated material, only in micrometer units, and, therefore, also a small amount of the material to be measured. For coarse-grained material or inappropriately oriented crystalline structure of the sample, significant errors may occur. Long computing time and high demands on the instrument and expertise of staff are also disadvantages.
- measurements with the feritscope is relatively quick and easy, if the sample surface is well prepared (ideally finely ground). The results are consistent and often they are in quite good agreement with the results of image analysis. The depth below the surface of the material in which the measurement is carried out is the greatest of the studied methods. However, this is only about 1-2 mm and, here too, with increasing thickness of the casting wall a slight increase of standard deviations values occurs. Therefore, if the size of austenitic and ferritic grains was greater than 1-2 mm, the results of the measurements could differ significantly. The measurement can also be adversely influenced by the presence of ferromagnetic phases other than a ferrite, e.g., some intermetallic phases.
- as the most appropriate method for measuring the ferrite content in castings of duplex stainless steels appears to be the image analysis of metallographic sample, or the measurement using the feritscope where it is necessary to take into account the grain size and possible presence of ferromagnetic phases other than ferrite. But when using any of the methods it is necessary to take into account the fact that the typical measurement error is not greater than 10 % so that to require a measurement precision of units or even tenths of a percentage point is unnecessary.

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